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Scalable, Low-Cost Deposition of Conductive Ni-Doped Polydopamine Thin Films for Electromagnetic Films

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Nicole M Ray[1,2]* and Alexander R Rouhani[2]

[1]School for Engineering of Matter, Transport and Energy, Arizona State University, 501 E Tyler Mall, Tempe, AZ, 85287, USA

[2]Rayn Innovations, 551 E University Dr, Tempe, AZ, 85281, USA

***Corresponding Author:** Nicole M Ray, School for Engineering of Matter, Transport and Energy, Arizona State University, 501 E Tyler Mall, Tempe, AZ, 85287, USA; Rayn Innovations, 551 E University Dr, Tempe, AZ, 85281, USA.

Abstract

Polydopamine (PDA) is a promising synthetic surface modifier that can be deposited on a wide range of substrates providing a chemically reactive surface with catechol and amine groups for functional coatings. In this study, bivalent metal ions were used to dope PDA for applications in functional conductive coatings. X-ray photoelectron spectroscopy (XPS) was used to verify doping in the PDA. To confirm doping in the matrix, the binding energy of characteristic peaks, shift in peaks and new signals indicating interactions between the dopant and PDA matrix were analyzed. Focused ion beam (FIB) cross-sectioning was used to measure the thickness and presence of the FeCo film layer demonstrating the doped PDA can be used for electrodeposition.

Introduction

Using examples from nature, new functionalized surfaces can be developed for applications in many different industries. Deriving from nature many scientific advances can be realized. One of the most influential discoveries within surface modification is the synthesis of polydopamine, derived from dopamine (DA) found in mussels [1]. Polydopamine (PDA) can be used as a surface modifier, adhesive, corrosion protection, primer layer, drug delivery, biosensors, etc. is one such material found in mussel foot proteins, inspiring use in functionalized materials. The pioneers of this work are Lee and Messersmith (2007), with research into mussel adhesive proteins for purposes in multifunctional polymer coatings [1, 2].

Polydopamine is a synthetic polymer inspired by the adhesive properties of DA, naturally occurring as neurotransmitter in the human body, a defense or signal compound in plants and metabolic intermediate in microorganisms. In recent years, PDA is used as an organic adhesive due to its unique properties and versatility. PDA has very strong adhesive properties arising from its catechol and amine groups in DA, which forms strong covalent bonds with a variety of surfaces, including metals,

ceramics, and polymers [2-4].

PDA forms through the alkaline oxidative self-polymerization of dopamine, in which DA is oxidized to dopamine-quinone, undergoes intramolecular cyclization, and subsequently crosslinks into a conformal polymeric network on virtually any substrate [1-3]. The resulting coating adheres strongly through covalent bonding, hydrogen bonding, π-π interactions, and metal coordination, enabling robust attachment even to hydrophobic or inert surfaces [1, 2, 5]. The film growth and adhesion strength depend strongly on pH, dopamine concentration, and oxygen availability, which regulate the kinetics of deposition and the resulting interfacial chemistry [4, 5].

In this research, the targeted application uses divalent metal ion doped PDA as a conductive thin film for applications in energy storage, sensors, biomedical devices, and advanced coatings. The specific method of depositing doped PDA film onto a substrate occurs via oxidative polymerization of dopamine followed by coordination/chelation of the metal ions into the polymer matrix. The divalent metal ions tested were copper, iron, and nickel.

Experimentation

For the use of an adhesion layer using PDA and the provided substrate, many iterations varying the concentrations, pH, time, buffer, and oxidation mechanism were varied. The process that was the most successful thus far includes using an autooxidation process with sodium periodate (SP) and Tris buffer [2, 5, 6].

The PDA solution was prepared by mixing 30-35 mM dopamine hydrochloride ($C_8H_{12}ClNO_2$) with 50 mM tris(hydroxymethyl)aminomethane ($C_4H_{11}NO_3$) in deionized water. The solution was stirred for 15 minutes, after which hydrochloric acid (HCl) was added to adjust the pH to above 8.0. Following the pH adjustment, sodium periodate ($NaIO_4$) was introduced at a 2:1 molar ratio of dopamine hydrochloride to sodium periodate. Three different metal dopants were added to the solution, copper sulfate [$CuSO_4{\cdot}5H_2O$], iron sulfate [$FeSO_4{\cdot}7H_2O$], and nickel sulfate [$NiSO_4{\cdot}6H_2O$], at a concentration level of 4 - 25 g/L. The mixture was stirred for another 15 minutes to initiate dopamine oxidation and polymerization into the polydopamine (PDA). Film deposition was carried out using a layered process. The first layer was formed by immersing UV-treated polymer or glass substrates into the PDA solution for 20 - 40 minutes, followed by rinsing with water [2, 4, 7, 8].

Results and Discussion

The experimental method successfully incorporated divalent metal cations into the PDA film. The film composition and surface morphology were analyzed using a NOVA 200 Focused Ion Beam (FIB). X-ray photoelectron spectroscopy (XPS), Kratos Axis Supra, was used to determine the elemental composition and chemical bonding behavior of the films. Varying concentrations of metal dopants were trialed as an attempt to develop a conductive PDA film. XPS analysis confirmed the incorporation of divalent metals from 1 - 3 at %, into the PDA matrix and verified the doping levels shown in Figure 1. The peaks for O 1s, C 1s, N 1s peak positions, dopant binding energies and atomic percent are found in the corresponding graphs shown in Figure 1. The nickel was doped at 1.17 at %, copper was doped at 2.49 at % and iron was doped at 2.31 at %. From the chemical composition data, it is shown that the divalent metal ions were in fact incorporated into the matrix as opposed to being loosely bound to the PDA surface. If the divalent metal was only loosely adsorbed into the PDA matrix the divalent metal ions would not show much change in C 1s, N 1s, or O 1s binding energies.

In Figure 1a, the Ni 2p binding energy is 853 eV, characteristic of Ni^{2+}, confirming the dopant presence. There are some trace contaminants (Na 1s and Si 2p) were also found in Figure 1a, caused from the glass substrate and residual salts from the solution. The PDA matrix ratios for C 1s, O 1s and N 1s align with the PDA composition in all scenarios and the structure is intact. Compared with the copper and iron dopant levels, the nickel content is slightly lower suggesting the nickel precursor has a lower solubility and may not bond as strongly for the PDA functional groups as the other bivalent metal ions tried. In Figure 1b, there is a clear doping presence stronger than Ni and Fe, with a binding energy of 932 eV. Note in this spectrum, there are elevated O and N suggesting the Cu is more interactive with the PDA's catechol and amine groups, favoring incorporation into the matrix. In Figure 1c, the Fe 2p peak plus broadened O 1s/N 1s peaks suggest the iron coordination is inside the PDA matrix rather than surface adsorption [8, 9].

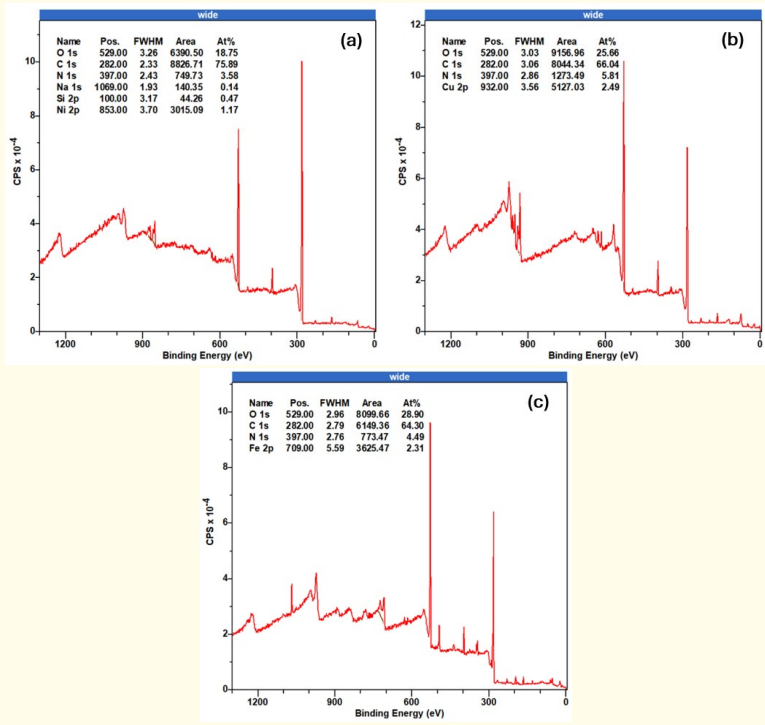

Figure 1: XPS graphs representing the spectrum of (a) 1.2 at % Ni, (b) 2.5 at % Cu, and (c) 2.3 at % Fe doped PDA.

The film appears as a thin black layer of deposit, coating the entire substrate, with the thickness ranging from about 0.02 - 20 µm depending on the number of layers. Figure 2 shows the Cu-doped PDA as a conductive intermediary layer. The sample has three total layers: (1) a layer of FeCo as a base, (2) a seed layer of Cu-doped PDA and (3) a top layer of FeCo. Based on the image and data, PDA is a viable seed layer for electrodeposition due to consistently successful depositions of the metal alloy film deposited on the doped PDA layers.

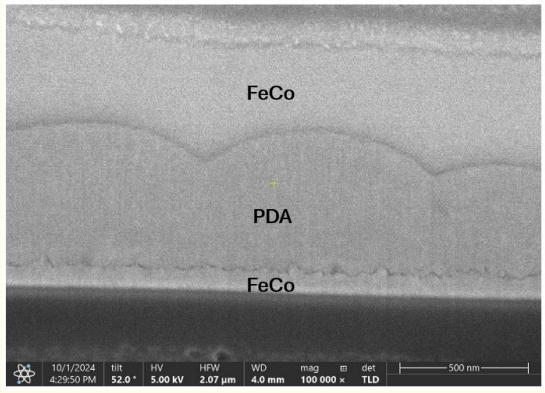

Figure 2: FIB cross section of single layer PDA film between two layers of electrodeposited FeCo alloy, using the doped PDA as a conductive layer.

An interesting effect of the PDA, with a thickness on average of 502 nm, as a seed layer for the electrodeposited metal alloy has a significant increase in deposition rate when depositing onto the PDA seed layer. For the sample shown in Figure 2, the two electrodeposited layers were deposited with identical conditions, with the first layer 84 nm thick (1.12 nm/s) while the second layer deposited on the PDA seed layer was on average 406 nm (5.41 nm/s) on average. Based on the data, the use of metals as a dopant with PDA results in an increased deposition rate compared with a sputtered copper conductive film.

The increased deposition rate arises due to doped PDA layer being rich in catechol, quinone, amine, and imine groups, while the incorporated bivalent metal ions provide metal-ligand coordination sites that act as high-energy nucleation centers. In contrast, a sputtered Cu/PET surface offers higher conductivity but fewer reactive sites. As a result, the doped PDA exhibits lower conductivity but significantly greater chemical reactivity, enabling denser nucleation, faster initial growth, and an overall higher deposition rate [9, 10].

Conclusion

In summary, the development of metal-doped PDA for applications in surface modifications, and conductive layers was successful for all three bivalent metal ions, with Cu-doped PDA having the highest efficiency and incorporation into the PDA matrix. While doped PDA is less conductive than copper sputtered films, the increase in chemical reactivity promotes rapid nucleation and growth. The resulting increase in deposition rate, combined with the versatility of PDA chemistry, highlights doped PDA films as a promising alternative for developing advanced conductive and functional coatings.

Compared to sputtered conductive films, PDA gives a more cost-effective approach because of many factors, including lower upfront costs such as equipment and operating costs. Sputtering requires high-vacuum, careful process control (pressure, temperature, gas flow, etc.), expensive targets, and cooling systems. Using a solution-based approach, as with the doped PDA, only basic coating and laboratory equipment is needed, the dopants are relatively inexpensive compared to sputtering targets, can deposit on many different substrates and can be easily scaled at a low cost.

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